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FACTORIAL DESIGN FOR THE ADSORPTION OF SOLID PARTICLES IN COAL EFFLUENT USING FEATHER-DERIVED ADSORBENT

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ABSTRACT

In the present study, batch adsorption of dissolved particles in coal effluent was examined. Analysis of the process was studied under 3-level full factorial design, with reference to three (3) factors viz: Mass of adsorbent, x_1(g), Contact time, x_2(mins), Volume of effluent, x_3(ml). In other words, 3^3-full factorial experiment was carried out. The average responses generated at various random experiments varied between 0.0525 – 0.065mg/l, and used to generate a linear model to fit the experimental data. The design expert computer software (7.0 version) was used to plot 3-dimensional surface relationship of the factors. It was observed, from the interaction plot of effluent volume versus adsorbent mass, that as the mass of adsorbent increases (with decrease in the volume of effluent), the uptake of the total solid particles rises. The case is different for the interaction of adsorbent mass and contact time, where more particles are being removed as both mass of adsorbent and contact time are increased. The variance (S_u^2) for the various number runs fall between 0 – 4.5×10^{-6}. The Chochrain's distribution (G) was calculated to be 0.3871. By simple comparison of $G_{calculated}$ with G_{table} (0.6798) at 0.05 level of significance, it was deduced that the hypothesis for the homogeneity of variance is acceptable. The t-test was, also, used to confirm the viability of the coefficients of the model equation, while the F-test was used to assess the adequacy of the model. The value of $F_{calculated}$ was found to be 0.7646, while that of F_{table} was 4.07 (>$F_{calculated}$), and this confirmed the adequacy of the model.

KEYWORDS: Factorial design, Adsorption, Solid particles, Coal effluent, Feather

INTRODUCTION

Design of Experiment (DOE) is the design of all information-gathering exercises, where variation is present, whether under the full or partial (fractional) control of the experiment. In testing, each experiment gets a minimum amount of measurable conversions, known as the sample size per data, for any statistical analysis (Billing, 2008). Therefore, the more experiment you have, the more conversions you require. The conversion data can also be thought as time, since the longer you leave your web page up, the more data you get. In an experiment, we deliberately change one or more process variables (or factors) in order to observe the effect (change) on one or more response variables (dependent variables). The design of experiments is an efficient procedure for planning experiments so that the data obtained can be analyzed to yield valid and objective conclusions. Hunter and Hunter (1998) reported that DOE begins with determining the *Objectives* of an experiment and selecting the *process factors* for the study. According to the report, an Experimental Design is the laying out of a detailed experimental plan in advance of performing the experiment. Well chosen experimental designs minimize the amount of information that can be obtained for a given amount of experimental effort. The Factorial Experiment is, thus, an experiment whose design consists of two or more factors, each with discrete possible values or *levels*, and whose experimental units take-on all possible combinations of the levels across all the factors (Maiti, 2001; Kashik, 2007). Brathy (1998) and Shilpi (2003), also, reported that a factorial experiment allows the study of the effect of each factor on the response variable, as well as the effects of the interactions between the factors on the response variable. During any experimental design, all possible interactions may be included in the test *(Full Factorial)*, or some of the interactions are selectively used *(Fractional Factorial)*.

For the vast majority of factorial experiments (Dick and Ewing, 1997; John, 2001; Box and Wilson, 1951), each factor is assigned two levels-*the high and low levels*. But for computational purposes, the factors may be scaled such that the high level is assigned a value of *+1*, and the low level is assigned a value of *-1*. In line with this arrangement, a full factorial design contains all possible combinations. Since full factorial gathers additional

information, it reveals all possible interactions, but there is always a "trade-off" when a higher number of factors is involved (≥5 factors); more data equals more information, but implies longer test duration with very high data requirements, since it shows all the experiments. On the other hand, the fractional factorial includes everything from only main effects to the interactions.

Frank Yates made significant contributions, particularly in the analysis of designs, by the "Yates Analysis" (Wikipedia, 1990). The *Yates analysis* exploits the special structure of the designs to generate least square estimates for factor effects, for all factors and relevant interactions. Maiti and Kenway (2004) reviewed that the analysis can be used to determine the ranked list of the factors, as well as the goodness-of-fit (as measured by the residual standard deviation) for the various models. The document unveiled that any given data should be arranged in "*Yates order*" before performing the Yates analysis. In this regard, given *'K-factors'*, the *'Kth'* column consists of $2^{(k-1)}$ minus sign (that is the low level of the factor), followed by $2^{(k-1)}$ plus sign (that is the high level of the factor). For instance, for a full factorial design with three factors, the design matrix is as follows:

-	-	-
+	-	-
-	+	-
+	+	-
-	-	+
+	-	+
-	+	+
+	+	+

And the Yates analysis generates the following output:

A factor identifier (from Yates order)- the specific identifier will vary, depending on the program used to generate the Yates analysis. Data plot, for instance, uses the following for a 3-factor model:
1 for factor 1
2 for factor 2
3 for factor 3
12 for interaction of factors 1 and 2
13 for interaction of factors 1 and 3
23 for interaction of factors 2 and 3
123 for interaction of factors 1, 2 and 3.

A rank list of important factors - this is the least square estimated factor effects ordered from largest in magnitude (most significant) to the smallest in magnitude (least significant).

A t-value for the individual factor effect estimates, which is computed as follows: $T = e/S_e$

Where e = the estimated factor effect
S_e = the standard deviation of the estimated factor effect.

The residual standard deviation that results from the model with the single term only, which is given by:

Response = Constant X $0.5x_i$ Where x_i = the estimate of the *i*th factor or interaction effect.

The cumulative residual standard deviation that results from the model using the current terms plus all the terms preceding the current term, and is given by:

Response = Constant + 0.5(all effect estimate down to t, including t).

This consists of a monotonically decreasing set of residual standard deviations (indicating a better fit as the number of terms in the model increases). The first cumulative residual standard deviation (*response = constant*) is for the model, and the constant is the overall mean of the response variable. The last cumulative residual standard deviation is given by:

Response = constant + 0.5 (all factor and interaction estimates).

It is important to note that if there are k-factors, each at 2 levels (*high and low*), then a full factorial design has 2^k runs (Perry et al, 1984; Sinnott, 1999). Following from this, when the number of factor is 5 or greater, a full factorial design requires a large number of runs, and is not very efficient. In this direction, the design guideline table recommends a functional factorial design or a plackett-Burman design for such case (Rose and Garba, 1991). The research work in course, thus, explores the content of this recommendation in the assessment of adsorption capacity of feather-derived adsorbent in wastewater management applications.

MATERIALS AND METHODS

Material Collection/Preparation

The wastewater sample (*coal effluent*) was collected from the effluent holding pond of the Coal Mining Industry, Akwukwe-Enugu, located at the South-Eastern part of Nigeria. The soil in the region is rocky with high coal deposit. Concentration of particulate matters in the coal washery effluent is always high as a result of its exposition to heavy down pours of rain.

The feathers of a Chicken were picked from the chicken-slaughter points at Obinze market in Imo State of Nigeria, sliced and impregnated in 60% strength of orthophospheric acid for 24-hours for activation. At the elapse of the time, the feather sample was removed and washed severally with distilled water to obtain a sample of neutral or nearly neutral pH (using a Delta-320 pH meter). The sample was then inoculated in clean trays and dried by means of oven dryer. It was, further, inoculated in clay pots and subjected to carbonization process at a charring temperature of 300^oC, using a furnace equipment of model- *2KX0-60*. A ceramic mortar set was used to grind the adsorbent sample (which is now in the form activated carbon), and a sieve size of *600μm* was used to obtain the granulated size that was used in the study.

The Factorial Experiment

Batch adsorption experiments were conducted randomly at three different levels – *the High level* (+1), *Low level* (-1) and *Base level* (0), with reference to three experimental factors – *Mass of adsorbent* (x_1), *Shaking/Contact time* (x_2) and *Volume of effluent* (x_3). Initial effluent concentration was checked by means of Ultraviolet Spectrophotometer (UVS) before the process. The finally concentration was, also, examined after every random treatment, and this served as response of the design process. Eight different experiments were conducted using the design array shown in Table 2 (Wikipedia, 1990), with replications to ascertain homogeneity of the experimental method.

RESULTS AND DISCUSION

The design expert computer software (7.0 version) was used to plot the 3-dimensional surface relationship of the factors – *Effluent volume* (ml), *Adsorbent mass* (g) and *Contact time* (mins.). It could be observed from the interaction plot of effluent volume versus adsorbent mass (Fig 1) that as the mass of adsorbent increases (with decrease in the volume of effluent), there is higher uptake of the total solid particles, unlike in figure 2, where more particles are being removed as both contact time and adsorbent mass increase. Figure 3 follow the same trend as Figure 1, that is as time increases (with decrease in effluent volume), there is more uptake of the particles. This suggests that low effluent volume implies less particle concentration in the solution.

Table 1: BASIS FOR THE FACTORIAL DESIGN

Factors	High Level (+)	Low Level (-1)	Base Level (0)
Mass of Adsorbent, x_1(g)	1.0	0.2	0.6
Shaken time, X_2(min)	10.0	2.0	5.0
Volume of Effluent, X_3(ml)	20.0	10.0	15.0

The proposed model equation is given by:

$$Y_u=b_0+b_1x_1b_2x_2+b_3x_3+b_{12}x_1x_2b_{13}x_1x_3+b_{23}x_2x_3$$

For each of the experiments (with replication, i.e. n=2), check for mean value of the runs (y), the variance (S_n^2) and the Chochrain’s distribution (G);

Where: $y_u = (y_1+y_1)/2$

$S_u^2 = \frac{1}{n-1} \sum_{K=1}^{n} (y_{uk} - y_u)^2$ and

$G = \frac{\text{Max. } S_u^2}{\sum_{u=1}^{n} S_u^2}$

These may be arranged in a summary table of array as follows:

Table 2: Design Array for Factor Responses

N	f_0 x_0	f_1 x_1	f_2 x_2	f_3 x_3	f_4 x_1x_2	f_5 x_1x_3	f_6 x_2x_3	Y_1(mg/l)	Y_2(mg/l)	$\bar{Y}_u$(mg/l)	S_u^2
1.	+1	+1	+1	+1	+1	+1	+1	0.055	0.056	0.0555	$2x10^{-6}$
2.	+1	-1	+1	+1	-1	-1	+1	0.050	0.055	0.0525	0
3.	+1	+1	-1	+1	-1	+1	-1	0.056	0.055	0.0555	$4.5x10^{-6}$
4.	+1	-1	-1	+1	+1	-1	-1	0.050	0.056	0.064	$2x10^{-6}$
5.	+1	+1	+1	-1	+1	-1	-1	0.055	0.054	0.065	0
6.	+1	-1	+1	-1	-1	+1	-1	0.051	0.055	0.064	$5x10^{-7}$
7.	+1	+1	-1	-1	-1	-1	+1	0.055	0.053	0.065	0
8.	+1	-1	-1	-1	+1	+1	+1	0.052	0.055	0.0635	$4.5x10^{-6}$
									$\sum S_u^2 = 4.65x10^{-5}$		

Thus, $G = \frac{4.5x10^{-6}}{1.1625x10^{-5}} = \mathit{0.3871}$

But G_{table} (0.05,8,1) = *0.6798*

Therefore, since G_{Table} = 0.6798>$G_{calculated}$ = 0.3871, the hypothesis for the homogeneity of variance is acceptable.

<u>NB</u>: The variance for the individual experiments is given by:

$S_E^2 = \frac{1}{8} \sum S_u \frac{1}{8} (4.65x10^{-5}) = \mathit{5.8125x10^{-6}}$

Evaluating the coefficients of the model equation:

$b_o = \frac{1}{8} (0.4315) = \mathit{0.0539}$

$b_1 = \frac{1}{8}$ (0.0555-0.0525+0.0555-0.053+0.0545-0.053+0.054-0.0535) = $\mathit{9.375x10^{-3}}$

$b_2 = \frac{1}{8}$ (0.0555-0.0525+0.0555-0.053+0.0545-0.053+0.054-0.0535) = $\mathit{6.25x10^{-5}}$

$b_3 = \frac{1}{8}$ (0.0555+0.0525+0.0555+0.053-0.0545-0.053-0.054-0.0535) = $\mathit{6.25x10^{-5}}$

$b_{12} = \frac{1}{8}$ (0.0555-0.0525-0.0555-0.053+0.0545+0.053-0.053+0.0535) = $\mathit{1.875x10^{-3}}$

$b_{13} = \frac{1}{8}$ (0.0555-0.0525+0.0555-0.053-0.0545+0.053-0.054+0.0535) = $\mathit{4.375x10^{-3}}$

$b_{23} = \frac{1}{8}$ (0.0555+0.0525-0.0555-0.053-0.0545-0.053-0.054+0.0535) = $\mathit{-6.255x10^{-5}}$

t_{table} = (8,0.05) = *2.3061*

$$S_b = \sqrt{\frac{5.8125x10^{-6}}{16}} = (3.6328)^{1/2} = 6.0273x10^{-4}$$

Thus, $(t_{table}) . (S_b) = (2.3061)(6.0273x10^{-4}) = 1.39x10^{-3}$

Comparing the value with coefficients of the model equation:
$(t_{table}) . (S_b) = 1.39x10^{-3} > b_2$, b_{12} and b_{23}; hence, they dropped from the model.

The model equation then becomes:
$Y_u = b_0 + b_1x_1 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3$
$Y_u = 0.0539 + 0.009375x_1 + 0.001875x_3 + 0.991875x_1x_2 + 0.004375x_1x_3$

Table 3: Checking for the Adequacy of the Model

N	1	2	3	4	5	6	7	8
$\hat{y}_u$	0.0714	0.0408	0.0677	0.0439	0.0598	0.0545	0.0552	0.0489
$\overline{y_u}$	0.0555	0.0525	0.0555	0.0530	0.0545	0.0530	0.0540	0.0535
$\hat{y}_u - \overline{y}_u$	0.0159	-0.0117	0.0122	-0.00091	0.0044	0.0015	-0.0012	-0.0046
$(\hat{y}_u\ \bar{y_u})^2$	$2.53x10^{-6}$	$2.53x10^{-4}$	$1.37x10^{-4}$	$2.28x10^{-5}$	$1.94x10^{-5}$	$2.25x10^{-5}$	$1.44x10^{-6}$	$2.116x10^{-5}$

SAMPLE CALCULATIONS

$\hat{y}_{u1} = 0.0539+0.009375+0.001875+0.001875+0.04375 = 0.0714$
$\hat{y}_{u2} = 0.0539+0.009375+0.001875-0.001875-0.04375 = 0.0408$
$\hat{y}_{u3} = 0.0539+0.009375+0.001875+0.001875+0.004375 = 0.0677$
$\hat{y}_{u4} = 0.0539-0.009375+0.001875+0.001875-0.04375 = 0.0439$
$\hat{y}_{u5} = 0.0539+0.009375-0.001875+0.001875-0.04375 = 0.0589$
$\hat{y}_{u6} = 0.0539-0.009375-0.001875-0.001875+0.004375 = 0.0545$
$\hat{y}_{u7} = 0.0539+0.009375-0.001875-0.001875-0.04375 = 0.0552$
$\hat{y}_{u8} = 0.0539-0.009375-0.001875+0.001875+0.04375 = 0.0489$
Then, $\sum(\hat{y}_u - y_u)^2 = 6.66605x10^{-6}$ (from the table).
Thus, $Q_L = 2x6.66605x10^{-6} = 1.3321x10^{-5}$
And $S_L^2 = \frac{Q_L}{\gamma_L} = \frac{1.3321x10^{-5}}{3}$ $4.444x10^{-6}$
Thus,

$$F = \frac{S_L^2}{S_E^2} = \frac{4.444x10^{-6}}{5.8125x10^{-6}} = 0.7646$$

But F_{table} (0.05, 8, 3) = *4.07*

Since $F = 0.7646 < F_{table} = 4.07$, the model is said to be adequate.

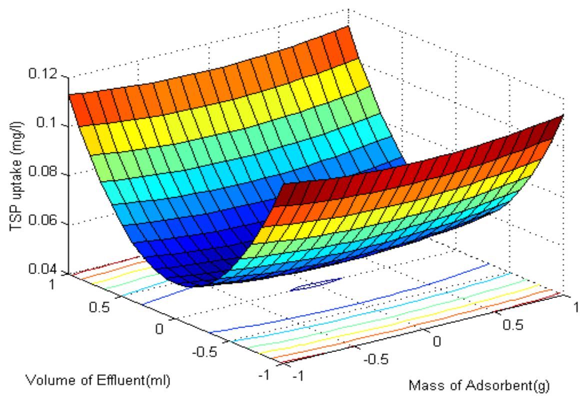

Fig 1: 3-D Surface Plot for Effluent Volume versus Adsorbent Mass

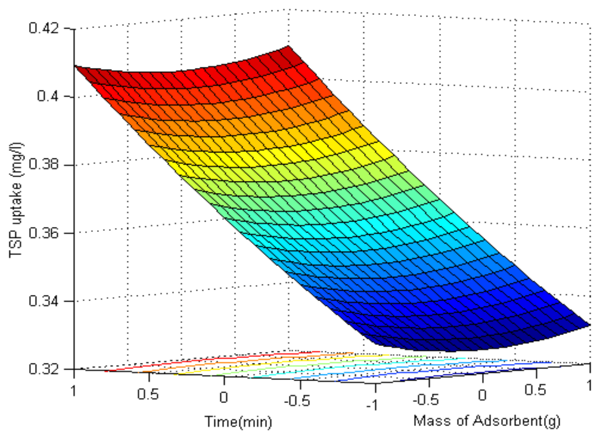

Fig 2: 3-D Surface Plot for Adsorbent Mass versus Time

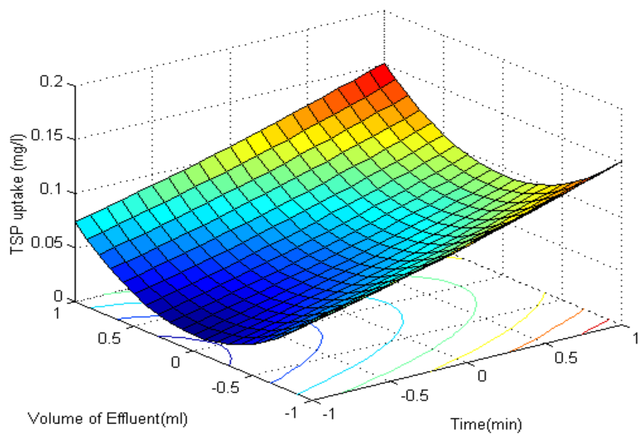

Fig 3: 3-D Surface Plot for Effluent Volume versus Time

CONCLUSION

Once the primary variables (factors) that affect the responses of interest are known, a number of additional objectives may be pursued. These include maximizing or minimizing a response, reducing variation and seeking

multiple goals. What each of these purposes has in common is that experimentation is used to fit a model that may permit a rough approximation to the actual requirement. In the present study, for instance, optimum average response (0.065mg/l) was recorded at runs 5 and 7; and the empirical model fitted the experimental data linearly.

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